



Anneal induced transformations of defects in hadron irradiated Si wafers and Schottky diodes

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A B S T R A C T

In this research, the anneal induced transformations of radiation defects have been studied in n-type and p-type CZ and FZ Si samples, irradiated with relativistic protons (24 GeV/c) and pions (300 MeV/c) using particle fluences up to $3 \times 10^{16} \text{ cm}^{-2}$. The temperature dependent carrier trapping lifetime (TDTL) spectroscopy method was combined with measurements of current deep level transient spectroscopy (DLTS) to trace the evolution of the prevailing radiation defects. The contactless TDTL technique has been shown to be preferential when the radiation induced trap density approaches or exceeds the dopant concentration and when it is necessary to avoid modification of a detector structure due to annealing processes at elevated temperatures. The deep level spectra were complementarily examined by using DLTS spectroscopy on Schottky diodes made of irradiated Si wafer fragments. The dominant radiation defects and their transform paths under isothermal and isochronal anneals have been revealed. A good agreement between the DLTS and TDTL spectra has been obtained.

1. Introduction

Silicon based detectors are widely used as particle detectors in experiments of high energy physics [1]. A deep understanding of radiation damage of particle detectors is important in order to extend the sensor lifetime and radiation hardness and potentially to restore their functionality after degradation caused by irradiation. A way to recover detector operational features is heat treatments at technically acceptable temperatures [1–3]. In order to develop adequate annealing procedures and technologies, it is crucial to characterize and understand the evolution of the most harmful radiation induced defects under heat-treatment procedures. Radiation technologies can also be useful for the manipulation of transducer switching rates [4]. However, radiation leads usually to an increase of leakage current and a degradation of barrier features [5]. Combining radiation and annealing technologies could develop into a promising tool for predictable improvements of transducer parameters.

In this work the combined study of radiation defects by deep level transient spectroscopy (DLTS) and temperature-dependent trapping lifetime (TDTL) techniques is used as an effective tool for the identification and tracking of radiation defects in heavily irradiated Si. There, application of standard spectroscopy techniques is limited or impossible

due to the extremely high densities of radiation defects significantly exceeding the concentration (N_D) of the shallow dopants. Therefore, the TDTL contactless technique, based on the measurements of the microwave-probed photoconductivity transients (MW-PC), was employed offering the advantage of excluding uncertainties due to the contact quality and disordered diode structure, due to the emerged clusters of radiation defects or even amorphous material regions. The correlation between the DLTS and TDTL spectral signatures enabled us to trace radiation defect transformations in the irradiated Si samples. The deep traps attributed to radiation defects in n-type and p-type Czochralski (CZ) and float-zone (FZ) grown Si were studied on various Si material wafer fragments and detector structures, irradiated with relativistic protons (24 GeV/c) and pions (300 MeV/c) using particle fluences up to $\Phi = 3 \times 10^{16} \text{ cm}^{-2}$. Variations of carrier recombination and trapping lifetimes were studied after isothermal anneals at 80 °C, varying heat treatment time from a few to thousands of seconds, and subsequent isochronal heat treatments for 24 h in nitrogen (N_2) ambient by discretely varied temperature from 80 °C to 300 °C. The temperature dependent variations of carrier recombination, trapping and emission lifetime were then measured after each heat treatment procedure. These latter measurements enabled us to compose a spectrum of prevailing radiation defects and to estimate their densities after each

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Table 1
Samples and irradiations.

Type of irradiation	Protons (p)	Pions (π^+)
Relativistic hadrons	24 GeV/c	300 MeV/c
Fluence range	$10^{12-3} \times 10^{16}$ p/cm ²	$10^{11-3} \times 10^{15}$ π^+ /cm ²
Si material	FZ n-Si CZ p-Si	CZ n-Si FZ n-Si
Dopant concentration/ resistivity	10^{12} cm ⁻³ 10^{12} cm ⁻³	10^{12} cm ⁻³ 10^{12} cm ⁻³
	> 3 k Ω cm 10 k Ω cm	> 3 k Ω cm > 3 k Ω cm

anneal step.

2. Experimental techniques and samples

2.1. Samples

Table 1 summarises the used materials and performed irradiations. Two sets of wafer fragments of the CZ (380 μ m thick) and float-zone (FZ) grown (280 μ m thick) n- and p-type Si of resistivity > 3 k Ω cm were irradiated with hadrons. The same irradiated-material wafer fragments were employed for Schottky diode fabrication. The Schottky barrier was formed by 30 nm thick Au evaporated layer on HF freshly-etched wafer top-surface. The ohmic contact was fabricated on the rear wafer side by evaporation of nickel (100 nm). An area of electrodes was set to 3.85×10^{-1} cm² for these samples to have proper barrier capacitance values and other reasonable parameters for DLTS measurements.

Irradiations by relativistic protons (24 GeV/c) and pions (300 MeV/c) were performed at CERN and at Paul Scherrer Institute in Villigen, respectively, varying fluences in the range of $\Phi_{p,\pi} = 10^{11-3} \times 10^{16}$ cm⁻². Fluence is presented as usual by denoting incident particles (protons (p) or pions (π^+)) within a beam cross-sectional area. The relativistic protons and pions induce depth-homogeneous distribution of radiation defects in samples of employed thicknesses, while the used range of fluences covers the actual interval of probable damage sustained by particle detectors in LHC experiments.

The isochronal anneals for 24 h have been performed at the temperatures in the range from 80 °C to 300 °C by temperature steps of 20–50 °C in N₂ gas ambient. The hadron irradiated samples were isothermally annealed at 80 °C up to 5 h before isochronal (24 h) anneals at elevated temperatures. The MCZ (Si grown by CZ with applied magnetic field) material was also examined for comparison of the recombination characteristics in the neutron as-irradiated wafer samples.

2.2. Spectroscopy techniques

2.2.1. DLTS spectroscopy of heavily irradiated samples

Deep level transient spectra over a temperature range of 15–300 K have been recorded on Schottky diode samples by using a HERA-DLTS System 1030 spectrometer [6]. The HERA-DLTS System 1030 spectrometer covers the scan temperature range from 10 to 450 K. The current (I) and optical (O) deep level transient spectroscopy (DLTS) measurements were performed. The DLTS transients were recorded by varying rate windows and using electrical carrier injection pulses of 100 ms duration and 9.9 V amplitude at reverse voltage of 10 V. The capacitance C-(O-)DLTS measurements were only employed for samples irradiated with the lowest fluences. The optical O-DLTS measurements were implemented using IR laser (1064 nm) pulses (of > 10 μ s). The measurements and data evaluation were controlled using the PhysTech software installed within the HERA-DLTS System 1030 spectrometer [6].

The barrier capacitance transient, which is determined by the majority carrier emission from deep levels with rate e_n (in n-type material), can be described by the relation

$$\frac{\Delta C(t)}{C} = \begin{cases} -\frac{1}{2} \frac{N_T}{N_{eff}} e^{-e_n t}, & e_n \gg e_p \\ 0, & e_n < e_p \end{cases} \quad (1)$$

Here, $\Delta C(t)$ is the barrier capacitance change due to the emission of carriers from a specific deep level, C is the barrier capacitance value under reverse bias for a fixed voltage, $e_{n,p}$ is the emission rate for electrons and holes, respectively. C-DLTS method is appropriate when the trap concentration N_T is less than 10% of the effective dopant (N_{Def}) concentration [7–11].

The current deep level transient spectroscopy (I-DLTS) method is preferable [7–11] when it is necessary to overcome limitation concerning effective doping in defect-rich samples. The transient I-DLTS signal of current density $\delta J(t)$ is then expressed as

$$\begin{aligned} \delta J(t) &= -\frac{1}{2} q x_d N_T \frac{e_n}{e_n + e_p} (e_n - e_p) e^{-(e_n + e_p)t} \\ &= \begin{cases} -\frac{1}{2} q x_d N_T e_n e^{-e_n t} (e_n \gg e_p) \\ \frac{1}{2} q x_d N_T e_n e^{-e_p t} \cong 0, (e_n < e_p) \end{cases} \end{aligned} \quad (2)$$

The amplitude of the transient signal δJ depends on the depletion width x_d , on the concentration N_T of deep traps and on the emission rate e_n . For the rather compensated materials, the optical excitation is commonly employed [7]. In this O-DLTS (Optical injection – DLTS) case, both majority and minority carriers determine the amplitude of the transient signal. The O-DLTS regime ($h\nu \leq E_g$) is close to that employed in photo-induced current transient spectroscopy (PICTS, $h\nu > E_g$) technique where transients are mainly governed by carrier density changes due to thermal emission.

2.2.2. Temperature dependent carrier trapping lifetime technique

Carrier thermal emission parameters can also be examined by the Temperature Dependent Carrier Trapping Lifetime (TDTL) method [12–15]. Contactless measurements of the temperature dependent carrier-trapping lifetime (TDTL) were performed by using a microwave probed photoconductivity transient (MW-PC) technique. The measurements were carried out using pulsed (400 ps) excitation at 1062 nm wavelength and a coaxial needle-tip microwave (22 GHz) probe within a near-field probing regime, performed by a proprietary measuring device VUTEG-6, fabricated at Vilnius University. Our proprietary TDTL instrument covers a scan temperature range from liquid nitrogen (~80 K) to 400 K. The combining of the DLTS and TDTL techniques in more detail had been discussed in our previous work [15]. This contactless TDTL technique is preferential when radiation trap density approaches or exceeds the dopant concentration. The two decay processes, recombination and (multi-) trapping-emission, can be distinguished when the two-componential (quasi-two-exponential) transients (Fig. 1(a)) are observed [14,15].

The models of carrier decay processes in the cases of several competing centers were considered in monographs [16,17]. A process of simultaneous recombination and trapping after short pulse excitation with initial ($t = 0$) excess carrier density $\Delta n(t = 0) = \Delta n_0$ can be described by a set of equations [17]:

$$\frac{\partial \Delta n}{\partial t} = -\frac{\Delta n}{\tau_R} - \frac{\partial \Delta m}{\partial t}, \quad (3A)$$

$$\frac{\partial \Delta m}{\partial t} = \gamma(N_{tr} - \Delta m)\Delta n - \gamma N_{CM} \Delta m, \quad (3B)$$

$$\frac{\partial \Delta p}{\partial t} = -\frac{\Delta p}{\tau_R}, \quad (3C)$$

$$\Delta p(t) = \Delta n(t) + \Delta m(t), \quad (3D)$$

$$\Delta p(t = 0) = \Delta n(t = 0) = \Delta n_0. \quad (3E)$$

Here, Δn , Δm and Δp denote the excess concentrations of free and trapped electrons as well as free excess holes, respectively, γ is the

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